



Phase Stability and Thermal Conductivity of Composite Environmental Barrier Coatings on SiC/SiC Ceramic Matrix Composites

Advanced environmental barrier coatings are being developed to protect SiC/SiC ceramic matrix composites in harsh combustion environments. The current coating development emphasis has been placed on the significantly improved cyclic durability and combustion environment stability in high-heat-flux and high velocity gas turbine engine environments. Environmental barrier coating systems based on hafnia (HfO_2) and ytterbium silicate, HfO_2 -Si nano-composite bond coat systems have been processed and their stability and thermal conductivity behavior have been evaluated in simulated turbine environments. The incorporation of Silicon Carbide Nanotubes (SiCNT) into high stability (HfO_2) and/or HfO_2 -silicon composite bond coats, along with ZrO_2 , HfO_2 and rare earth silicate composite top coat systems, showed promise as excellent environmental barriers to protect the SiC/SiC ceramic matrix composites.



Phase Stability and Thermal Conductivity of Composite Environmental Barrier Coatings on SiC/SiC Ceramic Matrix Composites

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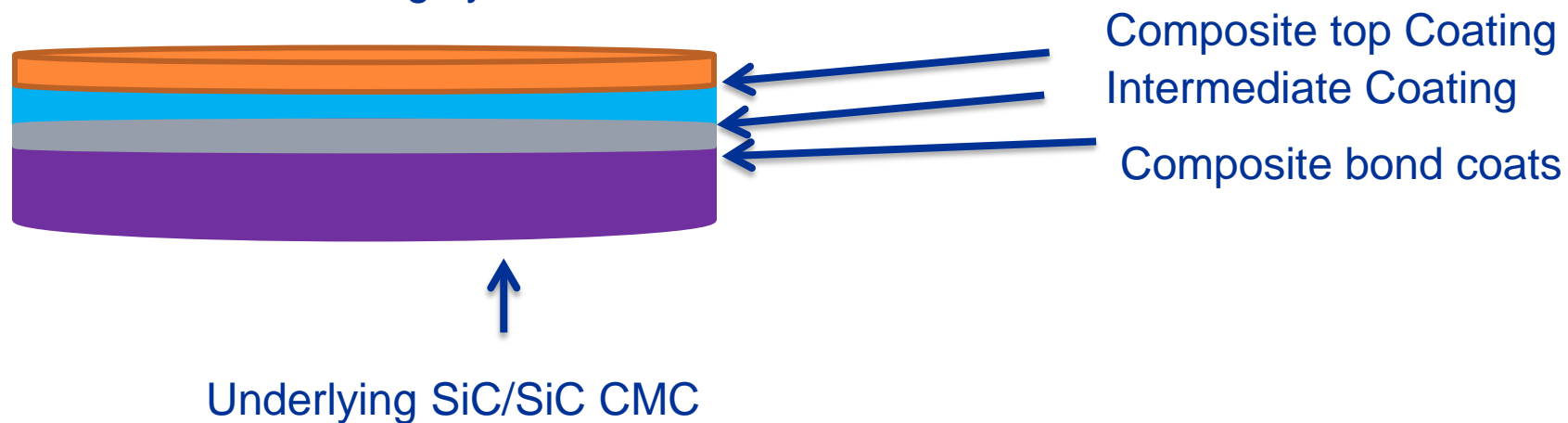
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Motivation

- Environmental barrier coatings (EBCs) for light-weight SiC/SiC ceramic matrix composite (CMC) components critical for advanced propulsion engines
- Higher temperature and higher strength capable coatings highly desirable
- Composite environmental barrier coatings have promise for improved temperature capability, stability and performance
 - High temperature stability is still of concern
 - $\text{HfO}_2\text{-Si}$ and $\text{HfO}_2\text{-SiCNT}$ bond coats as a major emphasis
 - $\text{HfO}_2\text{-Yb}_2\text{Si}_2\text{O}_7$ and/or $\text{HfO}_2\text{-Yb}_2\text{SiO}_5$ top coats
- Advanced coating systems were also tested





Outline

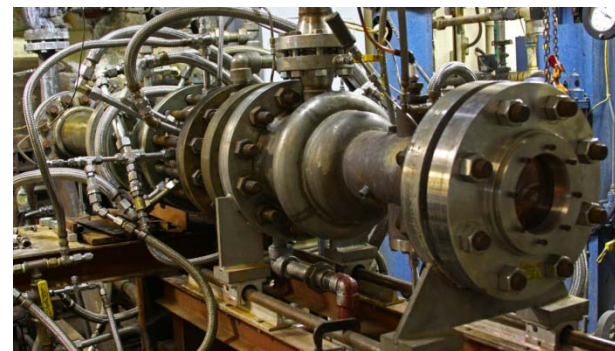
- Synthesis of Silicon Carbide Nanotubes (SiCNT)
 - Process optimization and scale up
 - TGA stability study in air
- Furnace Testing of Hot-Processed HfO_2 -Si and HfO_2 -SiCNT Composite Systems
 - Phase stability at 1400-1450°C
- Thermal Conductivity of HfO_2 -Si and HfO_2 -SiCNT systems
- High Pressure Burner Rig (HPBR) Stability of EBC-CMCs
- Cyclic Durability of Advanced Coating-CMC systems

Experimental

- SiC nanotubes synthesized by Si reaction with multiwall carbon nanotubes (CNTs) at 1400°C in Ar + H₂ environments
- Materials/specimens (hot-press): 1" diameter x ~3 mm thick HfO₂+Si and HfO₂+SiCNT disc specimens or coating HfO₂+Si and HfO₂+SiCNT specimens on CMCs
- Furnace stability testing in air by Thermogravimetric Analysis (TGA)
- X-ray diffraction and SEM characterization
- 3500 W continuous wave (cw) CO₂ laser (10.6 micron wavelength) used for high temperature thermal conductivity testing and high heat flux cyclic durability testing
- High Pressure Burner Rig Stability Testing



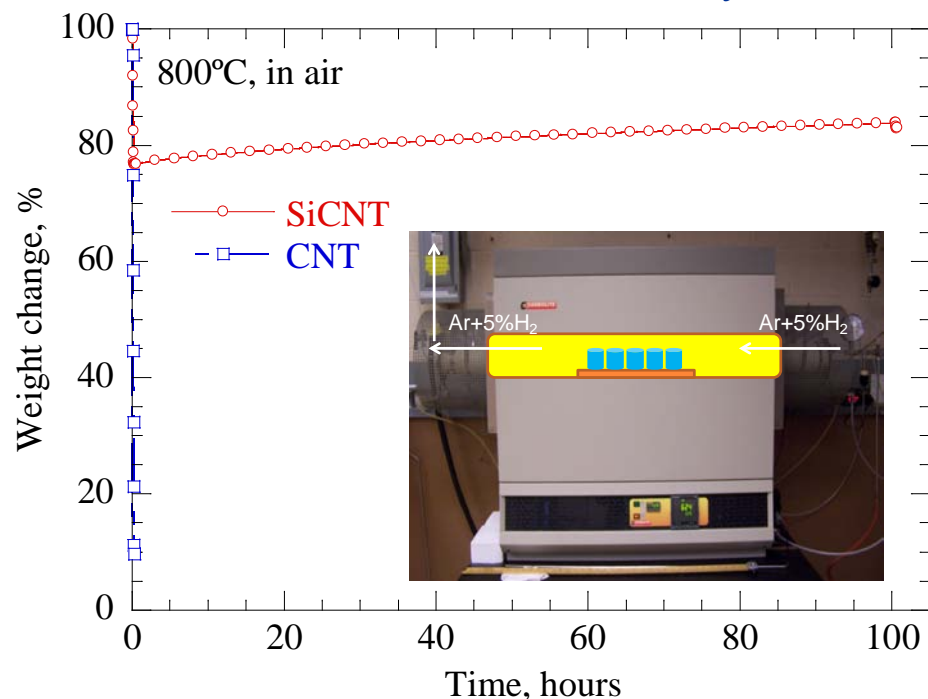
Laser test rig



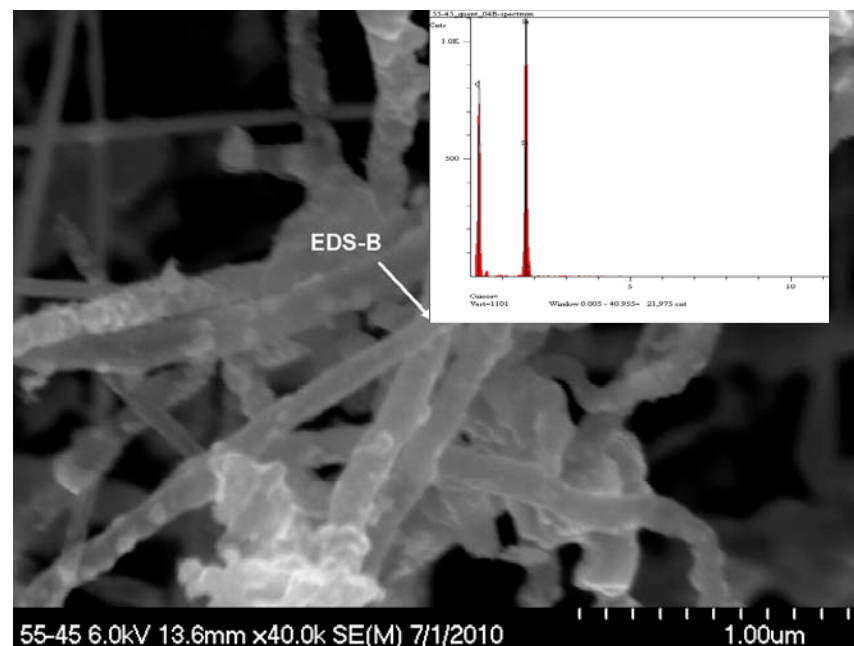
High Pressure Burner Rig

SiCNT Processing and Stability Testing

- Optimized SiCNT processing with Si to Carbon nanotube (CNT) weight ratio 55:45 for environmental barrier coating applications
- Obtained almost full β -SiC phase for the SiCNT based on X-ray diffraction
- Retained excellent nanotube morphologies based on SEM characterizations
- Scaled up the process with increased High quality SiCNT yields at 0.2 g/batch
- Demonstrated SiCNT stability in air using TGA

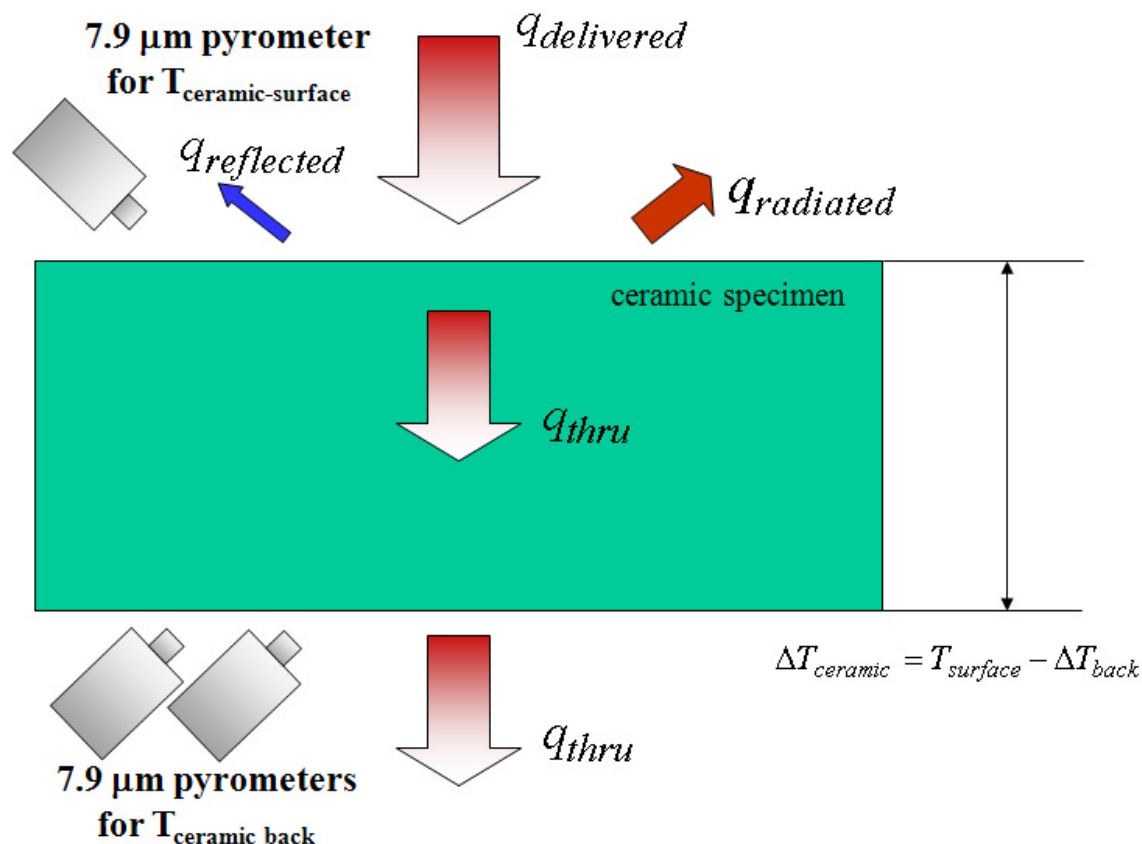


SiCNT processing and TGA stability evaluation

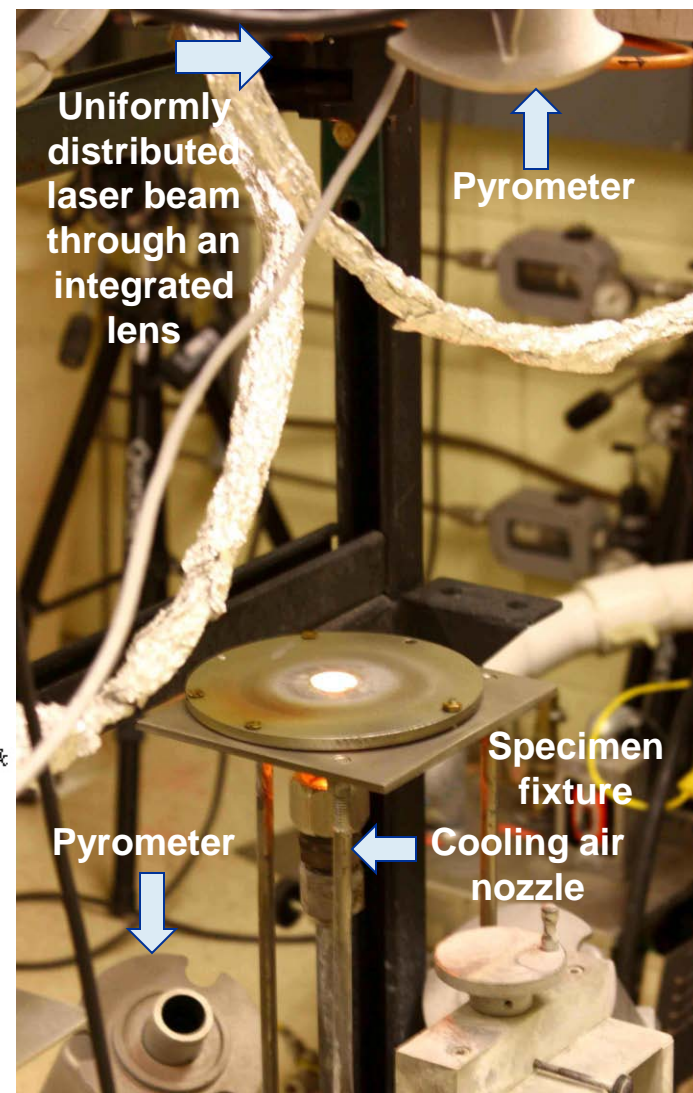


SEM and EDS characterizations of SiCNTs

Laser Steady-State Heat Flux Thermal Conductivity Testing Approach



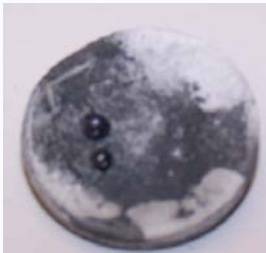





Schematic of the laser heat flux test principles



Furnace Testing of Hafnia-Silicon Systems

- Optimum mixture composition found between 25wt% and 50wt%Si

Temperature	HfO ₂ +25%Si	HfO ₂ +50%Si	HfO ₂ +75%Si
1350°C			
1400°			
1450°C			

The HfO_2 +SiCNT Bond Coats Showed High Temperature Stability in Oxidizing Environments

- HfO_2 , SiCNT and HfO_2 silicate are major phases after the 100hr testing

Before Testing



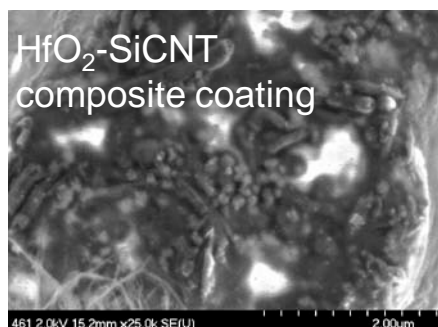
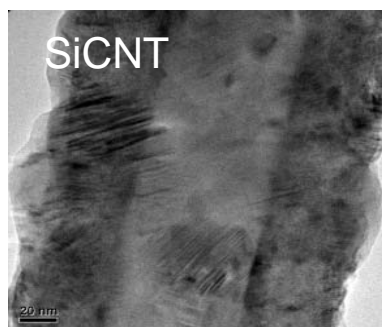
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1400°C/50hr

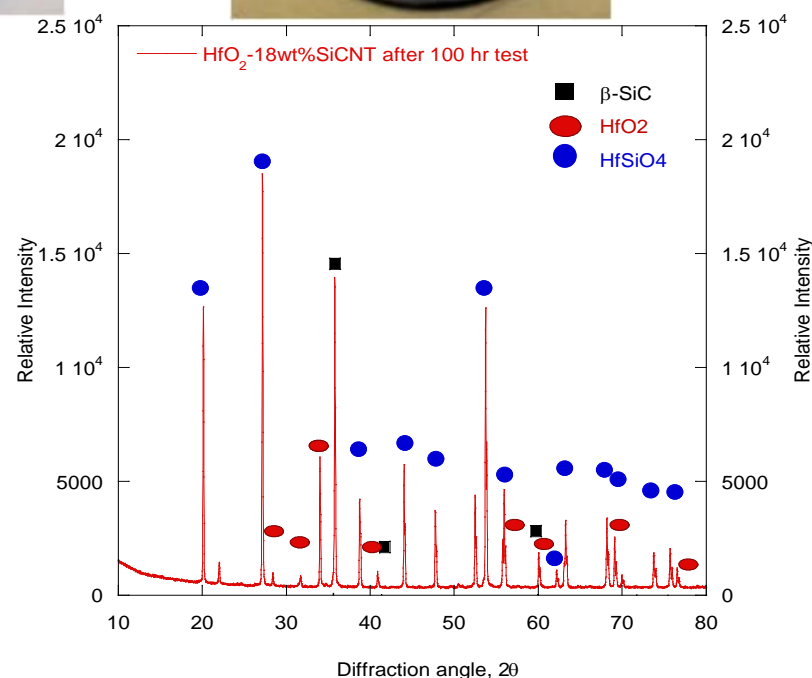


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1450°C/50hr

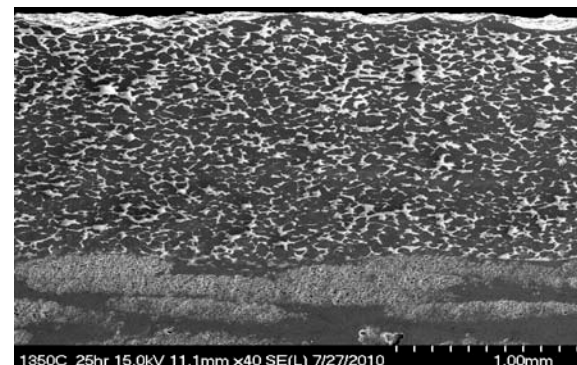
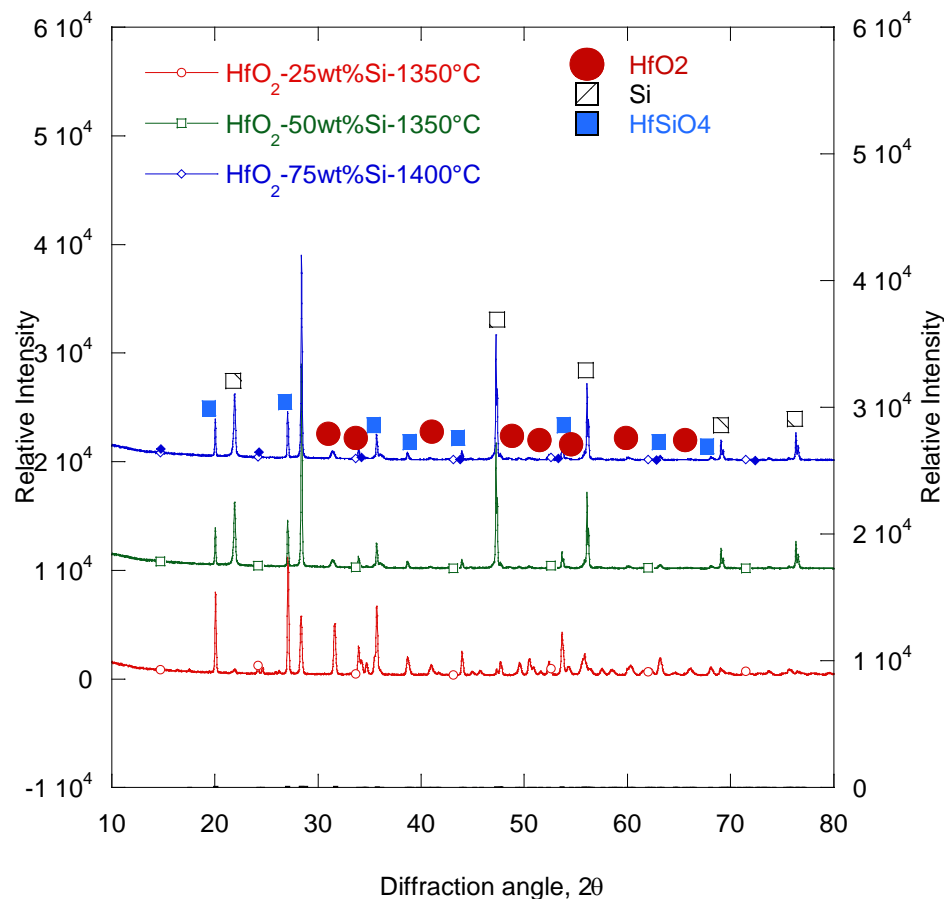


SiC nanotube (SiCNT) and HfO_2 -based nano-composite coatings



The HfO_2 +Si Bond Coats Showed Significantly Improved Temperature Capability

- Higher stability observed even after 1450°C testing



Cross-section, Hot-pressed HfO_2 -50wt%Si on CMC, 1350°C tested



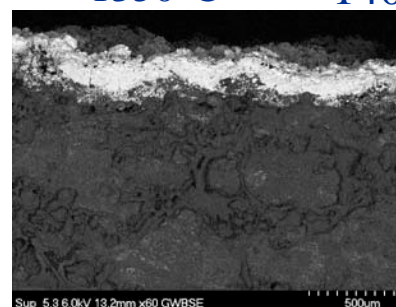
1350°C



1400°C



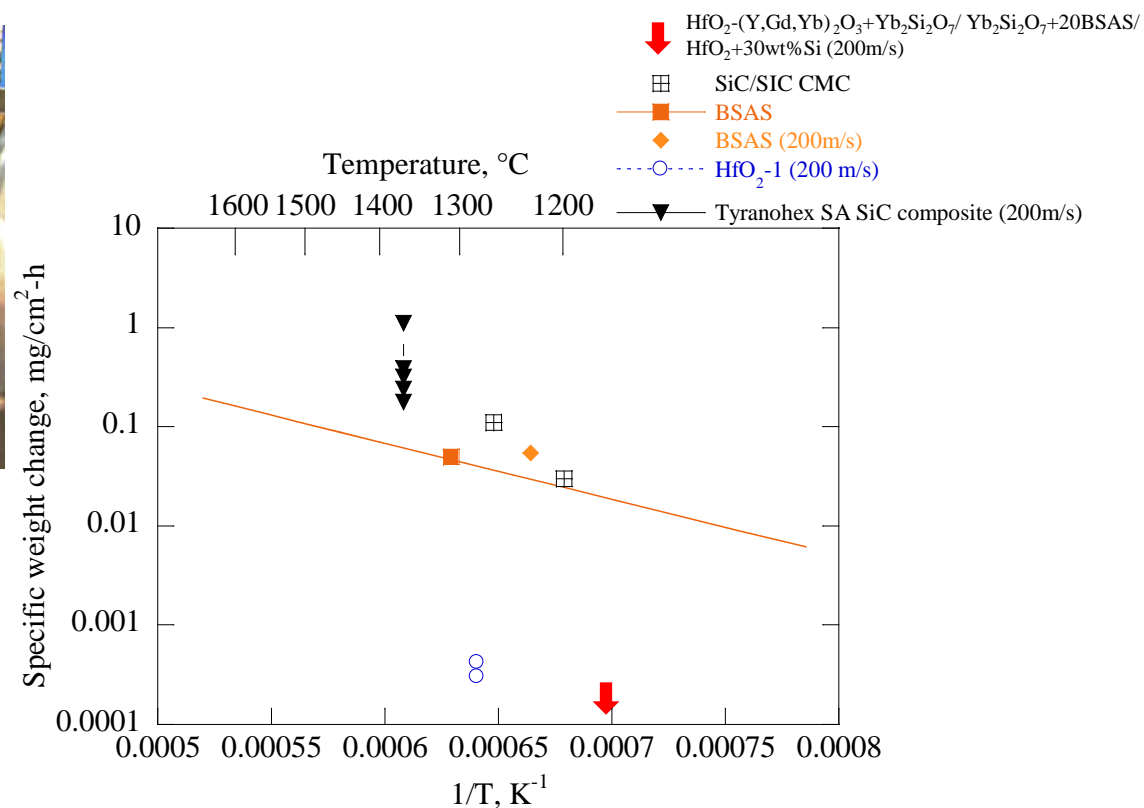
1450°C



HfO_2 - HfO_2 +30Si
bond coat on CMC,
1500°C tested for 200
hours

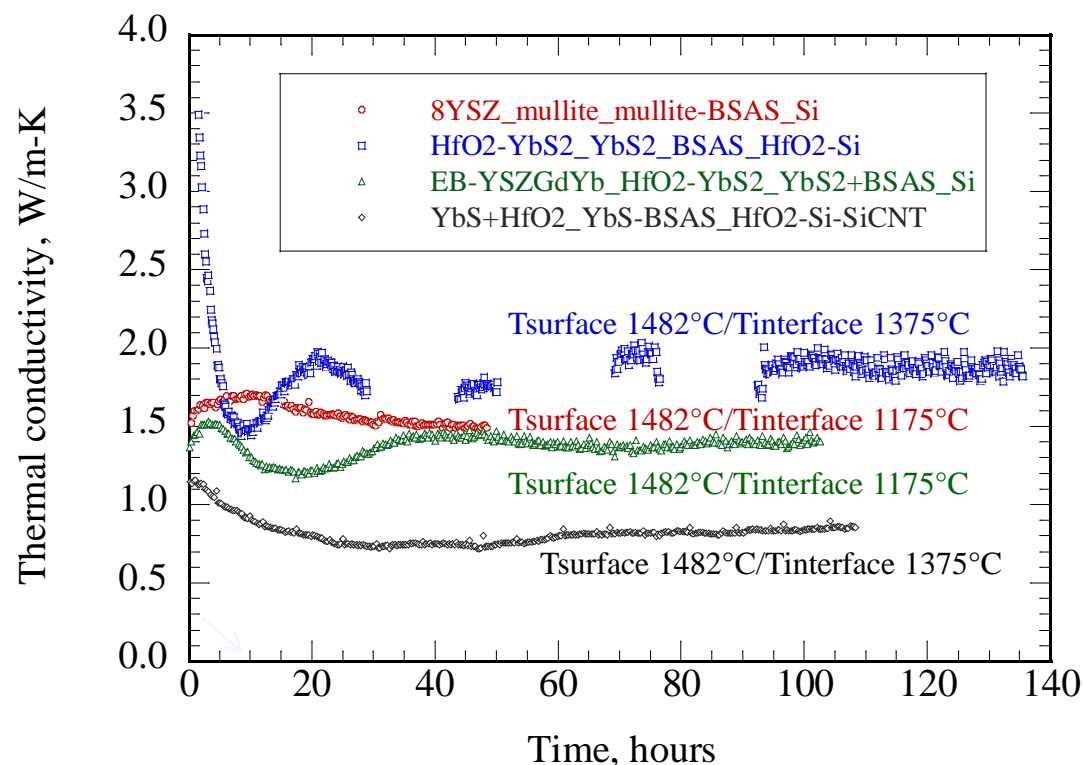
High Pressure Burner Rig Recession Testing

- HfO₂/ytterbium silicate/HfO₂-Si composite bond coat tested at 200 m/s in high pressure burner rig and showed excellent stability



High Heat Flux Thermal Gradient Cyclic Durability Evaluation of Environmental Barrier Coating and Bond Coat Systems

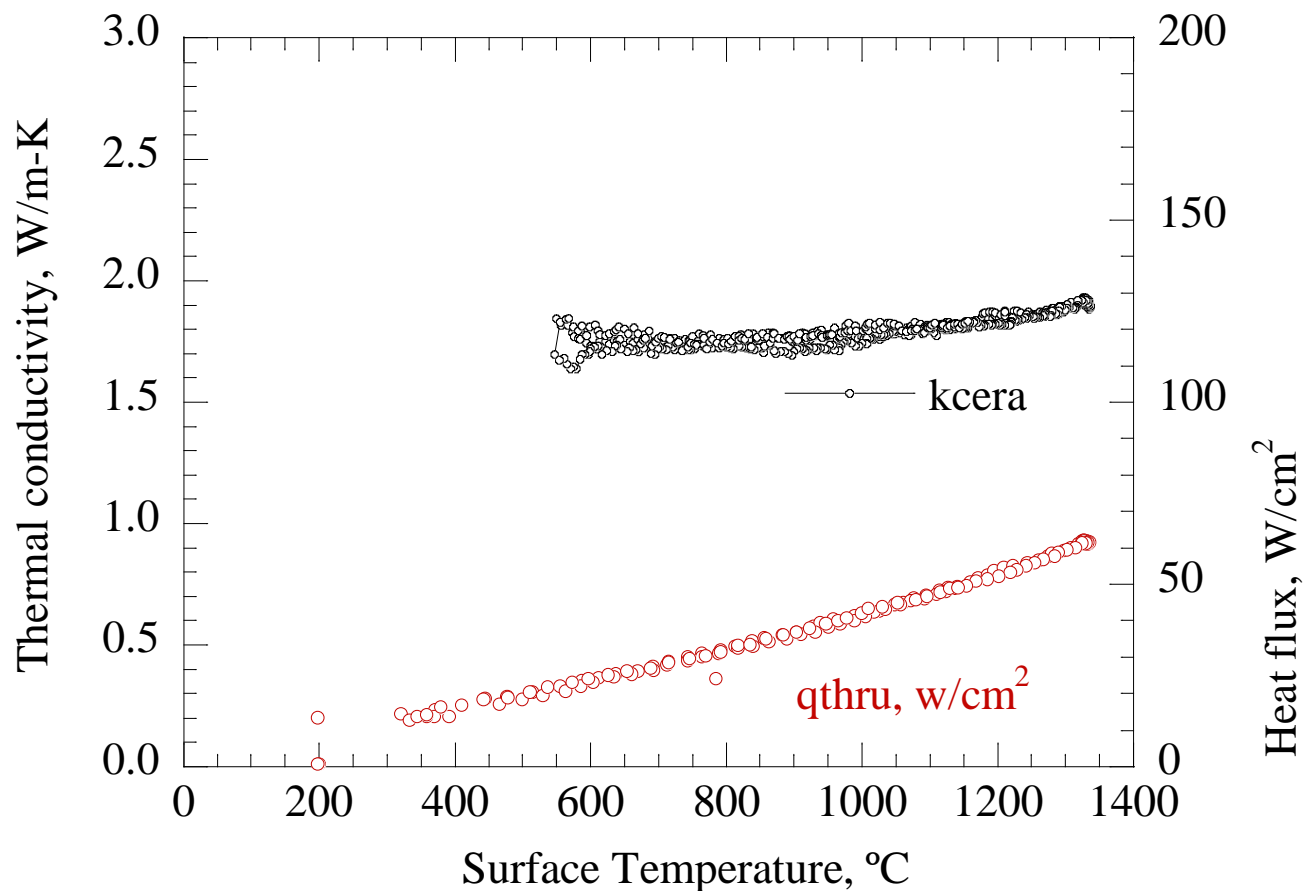
- Laser high heat flux rig thermal gradient thermal cyclic testing at surface temperature of 1482°C (2700°F)
- Advanced bond coats tested at up to 1375°C



HfO₂-ytterbium silicate coating
with HfO₂-Si bond coat, after
100hr testing at T_{surface}
1482°C/T_{interface} 1375°C

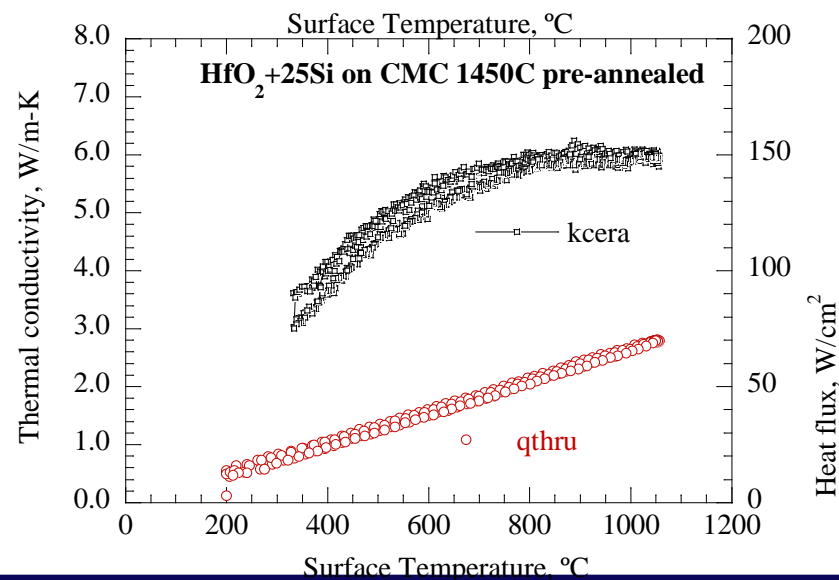
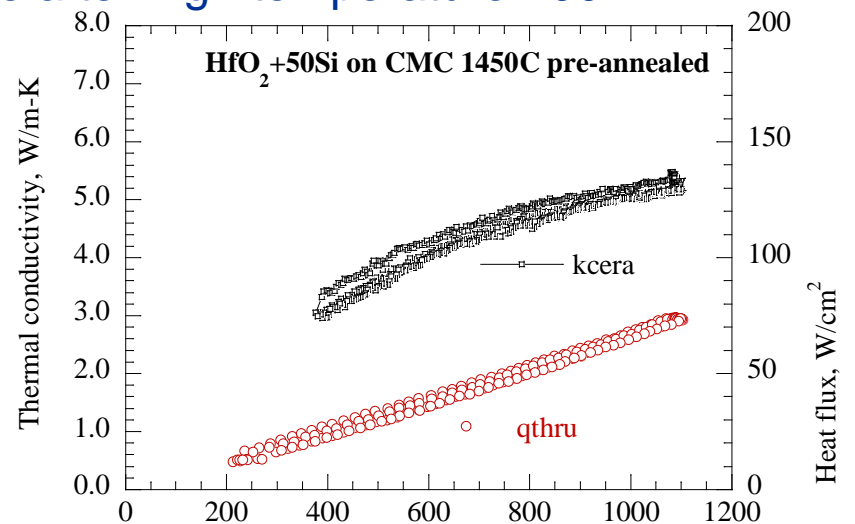
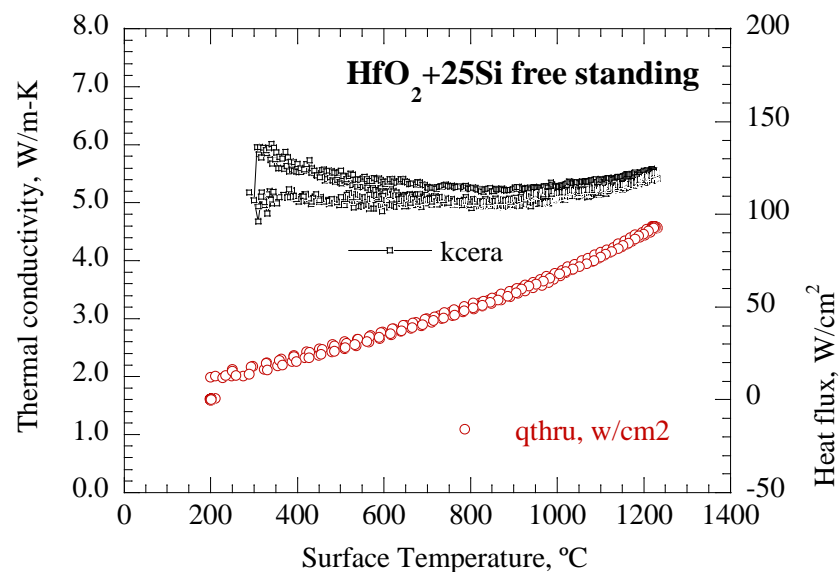
Thermal Conductivity of HfO_2 -12.4wt%SiCNT

- Effect of nanotube fractions on thermal conductivity is being evaluated and modeled



Thermal Conductivity of HfO_2 -Si Systems

- Thermal conductivity maintained stable after high temperature 100 hr annealing





Summary

- Composite HfO_2 -Si and HfO_2 -SiCNT bond coats synthesized for high temperature environmental barrier coating applications
- The stability of HfO_2 -Si and HfO_2 -SiCNT bond coats demonstrated for stability up to 1450°C (1500°C for advanced processed coatings)
- Thermal conductivity of HfO_2 -Si and HfO_2 -SiCNT bond coats evaluated
- Multilayer EBC composite coatings showed combustion gas stability and thermal gradient durability in high pressure burner rig and laser high heat flux rig



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